IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:	Gehrke, et al.	Confirmation No. 7739
Serial No.:	10/763,588	
Filing Date:	January 23, 2004	
Examiner:	M. Guerrero	
Art Unit:	2822	
Attorney Docket:	013921-000026	
Title:	Pendeoepitaxial Methods of Fabricating Gallium Nitride Semiconductor Layers on Sapphire Substrates, and Gallium Nitride Semiconductor Structures Fabricated Thereby	

Commissioner of Patents
Post Office Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R § 1.131

We, Thomas Gehrke, Kevin J. Linthicum, and Robert F. Davis, do hereby declare that:

1. We are the applicants of the above-identified patent application and its priority applications, and co-inventors of the subject matter described and claimed therein.

2. Between the dates of November 17, 1997 and October 3, 1999, while working in the United States, we conceived the idea of fabricating gallium nitride semiconductor structures by using pendeoepitaxy to fabricate gallium nitride semiconductor layers on sapphire substrates as described and claimed in our applications. A notebook page, documenting a meeting wherein the invention was discussed, was prepared and signed by two of us, Thomas Gehrke and Kevin J. Linthicum, in the course of working on the invention. The notebook page contains two sketches, which were prepared and included in the notebook page prior to the two of us executing the page. A copy of the notebook page is attached hereto as Exhibit A and all dates on the Exhibit have been redacted. The date on which the notebook page was executed falls between November 17, 1997 and October 3, 1999. The sketches substantially correspond to Figures 4 and 9 within the above-identified patent application.

3. Subsequent to the preparation of the notebook page described above, but still prior to October 3, 1999, an Email was sent by one of us, Thomas Gehrke, to outside patent attorney

Mitchell S. Bigel, suggesting figures for the proposed patent application. The proposed figures were attached to the Email in the form of a Microsoft PowerPoint™ file. Copies of the Email and accompanying figures are attached hereto as Exhibit B and all dates on the Exhibit have been redacted. The date stamp on the Email falls between the date of the notebook page described above and October 3, 1999. Figures 1-6 attached to the Email substantially correspond to Figures 17-22 within the above-identified patent application.

4. Subsequent to the Email described above, but still prior to October 3, 1999, an invention disclosure form was prepared, signed, and dated by all of us in the course of working on the invention. A copy of the invention disclosure form is attached hereto as Exhibit C and all dates on the Exhibit have been redacted. Additional information not related to the conception, nature or description of the invention has also been redacted. The date on which the disclosure form was executed is subsequent to the date stamp on the Email described above, but still prior to October 3, 1999. The descriptive material in paragraphs 6-10 of the invention disclosure form coincides with descriptive material within the above-identified patent application.

5. Subsequent to our preparation of the invention disclosure form described above, it was provided to North Carolina State University's Office of Technology Transfer and Industry Research (NCSU-OTTIR).

6. On information and belief, subsequent to our preparation of the invention disclosure form described above and its provision to North Carolina State University's Office of Technology Transfer and Industry Research, a letter was sent from Dr. David Winwood of North Carolina State University to outside patent attorney Mitchell S. Bigel, requesting that the priority patent application be prepared and filed in the U.S. Patent and Trademark Office. A copy of this letter is attached hereto as Exhibit D, and all dates, as well as references to matters not related to the present declaration, have been redacted. The date on the letter is subsequent to the date on the invention disclosure form described above, but still prior to October 3, 1999.

7. Between the dates of October 5, 1999 and November 12, 1999, an initial draft of the patent application was sent from outside patent attorney Mitchell S. Bigel, to one of us, Kevin J. Linthicum, asking that all of us review the draft for technical accuracy and advise Mr. Bigel

of any suggested changes or corrections. A copy of the cover letter accompanying this draft is attached hereto as Exhibit E, and all dates have been redacted. The date on the letter falls between October 5, 1999 and November 12, 1999.

8. Two of us, Thomas Gehrke and Kevin J. Linthicum, executed the declaration of inventorship for the priority application corresponding to the above-identified application on November 12, 1999, while the third, Robert F. Davis, executed the declaration of inventorship on November 17, 1999. The declaration of inventorship is on file at the U.S. Patent and Trademark Office with the above-identified application.

9. The priority patent application corresponding to the above-identified patent application was filed on February 17, 1999.

We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

This declaration was executed by each of us on the dates indicated.

_____ Date: 02/25/06, 2006
Thomas Gehrke

_____ Date: 03/09/06, 2006
Kevin J. Linthicum

Robert F. Davis Date: 03/21/06, 2006
Robert F. Davis

Follow up meeting: Thomas
Kevin
[redacted]

Pendeo Epitaxy on Sapphire

problems:

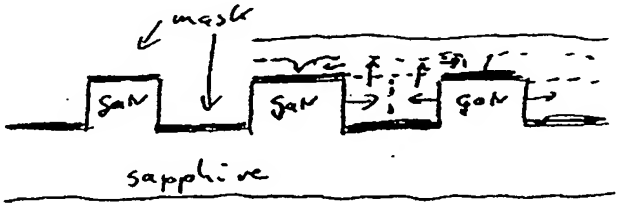
1) nucleation in the trenches, see results of Tg-450
2) GaN, which nucleated in the trenches, is interfering with laterally grown GaN from the posts/stripes

good news:

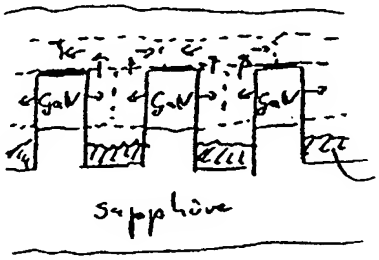
1) lateral growth off the sidewalls visible
2) stripe orientation, as determined by Laue-experiment correct

solutions/ideas:

1) put mask down in the trenches, like the mask on the posts, no GaN will nucleate on the mask at elevated growth temp.:



2) etch deep trenches and put posts close together, lateral growth will coalesce before it interferes with nucleated growth in trenches



↳ question: how narrow can we make posts?

[redacted] [redacted]

To: mbigel@carolinapatents.com
From: Thomas Gehrke <tgehrke@eos.ncsu.edu>
Subject: pendeo-epitaxy on sapphire
Cc:
Bcc:
X-Attachments: :People's Folders:8950:PEonsapphire.doc: :People's Folders:8950:PEonsapFIG.ppt:

Hi Mitch,

just in case if you would like to take a look on what I have for now regarding the pat. appl. for pendeo-epitaxy on sapphire.
Attached to this e-mail is a word document 'PEonsapphire.doc' and the figures 'PEonsapFIG.ppt' as a PowerPoint file. Please let me know what you think and what you think is the best way to proceed.

Thank you very much,
Thomas

FIG. 1

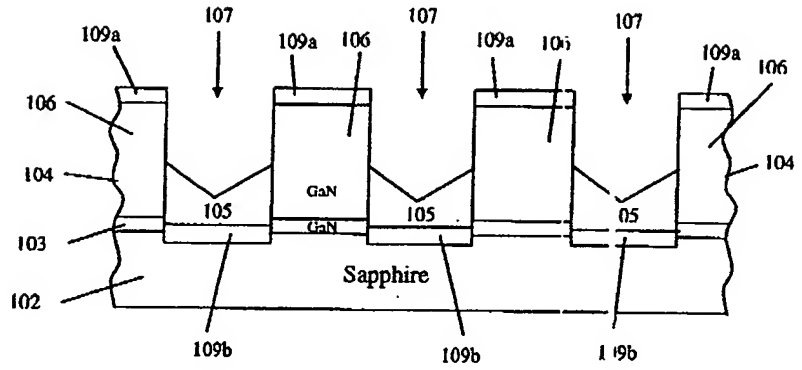

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FIG. 2

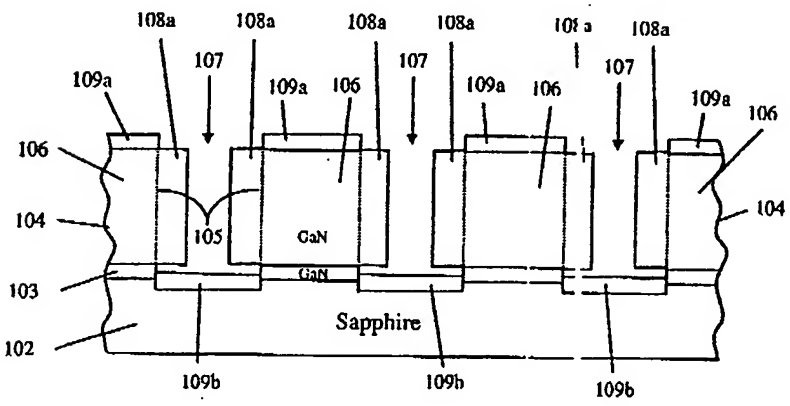
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FIG. 3

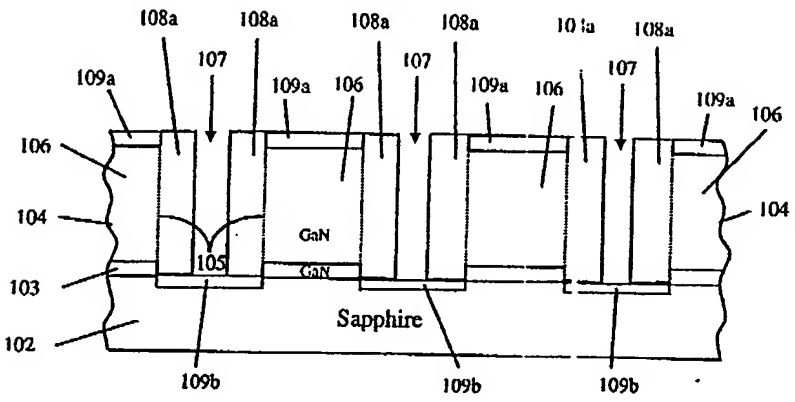
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FIG. 4

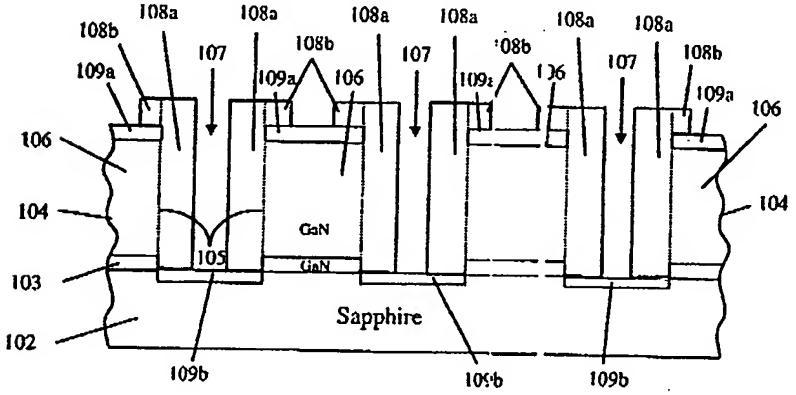
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FIG. 5

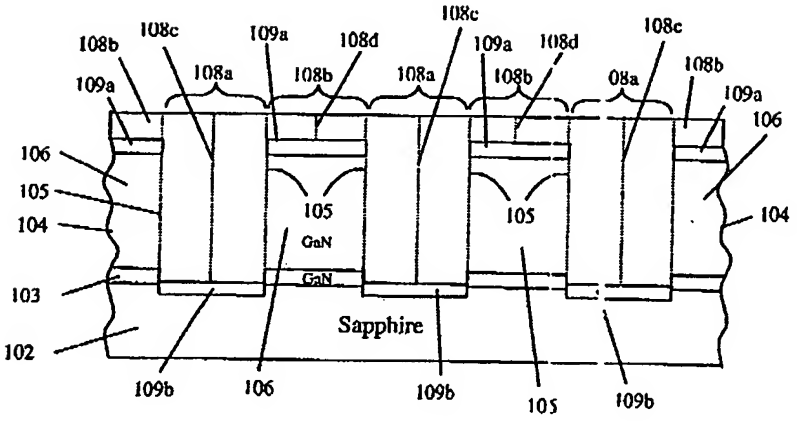
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FIG. 6

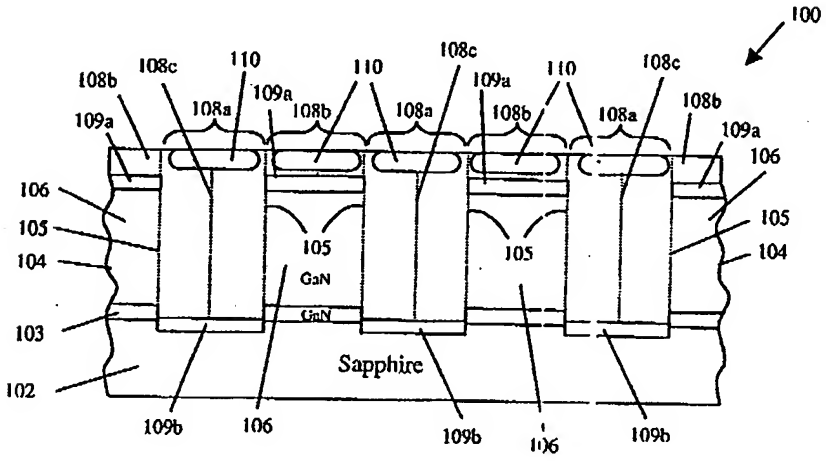
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FIG. 7

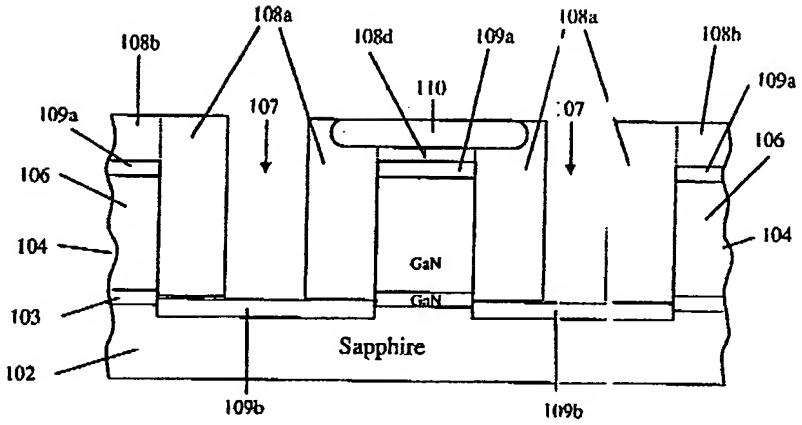
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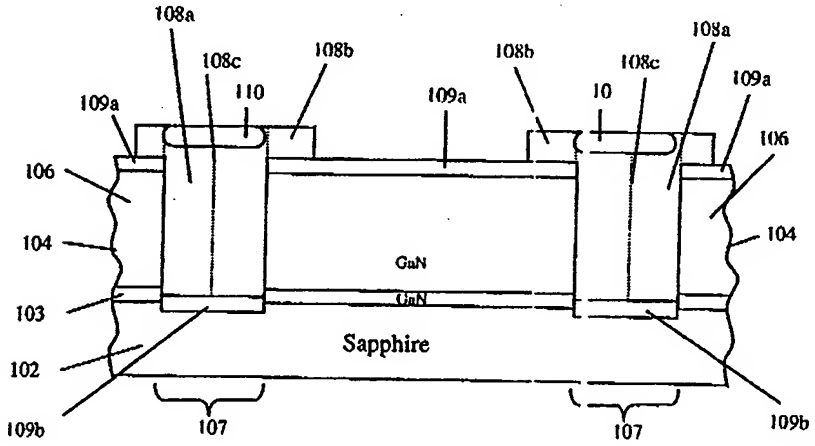
FIG. 8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CONFIDENTIAL (NCSU Patent Office Use Only)

NCSU File No. 99-79

Lawyer's File No. _____

NCSU INVENTION DISCLOSURE FORM

This form must be signed by the Department Head and the College Dean/Associate Dean prior to submission.

Inventor's Name: Thomas Gehrke
Title: Graduate Research Assistant
Mailing Address: Box 7907, NCSU,
Raleigh, NC 27695 - 7907

Citizenship: Germany
Dept: Materials Science & Engineering
Tel: 919 515 6619

Inventor's Name: Kevin James Linthicum
Title: Graduate Research Assistant
Mailing Address: Box 7919, NCSU,
Raleigh, NC 27695 - 7919

Citizenship: USA
Dept: Materials Science & Engineering
Tel: 919 515 6177

Inventor's Name: Robert F. Davis
Title: Professor
Mailing Address: Box 7907, NCSU,
Raleigh, NC 27695 - 7907

Citizenship: USA
Dept: Materials Science & Engineering
Tel: 919 515 3272

1. **Title of Invention:** Process for Growing Low Defect Gallium Nitride Semiconductor Layers on Sapphire Wafers Using Lateral Growth From Sidewalls, and Gallium Nitride Semiconductor Structures Fabricated Thereby

2. Date Invention Conceived (Conception Date): [redacted]

3. Has the Invention been reduced to practice? Yes, the concept of pendeo-epitaxy on the substrate sapphire has been reduced to practice on [redacted]

4. Supporting Data Notebook: [redacted]

5. Has the Invention been published: Orally: No In Writing: No

6. Brief Description. Is the Invention a new process, composition of matter, a device or one or more products? A new use for, or an improvement on an existing process? Please Describe.

   The invention is a new process for producing single crystal gallium nitride (GaN) semiconductor layers having a low density of line and planar defects using single crystal sapphire (Al_2O_3) wafers. This is accomplished by utilizing a transition structure between the single crystalline sapphire and the single crystalline GaN. The transition structure is comprised of an initial GaN "seed" layer grown using standard OMVPE growth techniques.

   Next, portions of the GaN seed layer are completely etched away to the surface of or into the sapphire substrate resulting in the formation of columnar structures of the GaN seed layer (and more importantly, a plurality of exposed GaN seed sidewalls). The top surface of the GaN seed columns may or may not be capped with a mask material.

   Finally, a pendeo-epitaxial regrowth of GaN is produced by lateral growth of GaN from the sidewalls of the GaN seed columns, vertical GaN growth from the extending GaN lateral growth, and the subsequent lateral growth over the unmasked or masked top of the column utilizing the conventional lateral epitaxial overgrowth (LEO) technique. Both the pendeo-epitaxial and LEO growths continue until they coalesce with the adjacent thin film growth, thereby forming a continuous layer of low defect density GaN and subsequent GaN semiconductor structures fabricated thereby.

7. Write a brief descriptive abstract of your Invention without making a disclosure. This will be used for marketing purposes.

   The invention is a process of fabricating high-quality low-defect single-crystalline GaN semiconductor layers on single-crystalline sapphire substrates through the use of a transition structure comprised of pendeo-epitaxial grown GaN via deposition by standard vapor deposition and epitaxial growth techniques. Typical defect densities of $<10^5$ cm^{-3} over the entire GaN surface are achieved. The area of the low-defect GaN layer is only limited by the size of the substrate. This is a significant improvement over the conventional LEO technique where low-defect density GaN growth is limited only to areas of overgrowth regions, or requires multiple LEO iterations to achieve equivalent results to this single-step regrowth process.

8. From the description, pick out and expand on novel and unusual features. How does the Invention differ from present technology? What problems does it solve or what advantages does it possess?

Single crystal semiconductor layers having low densities of line and planar defects are required for microelectronic and light-emitting devices having optimum performance. Typically, epitaxial thin films contain all the defects of the underlying buffer layers and/or substrates on which they are grown. Additionally, since currently there are no "bulk" GaN substrates commercially available for homoepitaxial growth, GaN heteroepitaxial growth is required and results in further generation of misfit and threading dislocations and occasional planar defects in the GaN layers. Recent advances in growth of GaN on sapphire via LEO growth techniques has resulted in a reduction in defect densities of approximately 10^5 cm^{-3}. However, the growth of this low defect density GaN is limited to the areas over the masked regions in the LEO growth.

This invention allows for the fabrication of low-defect density ($\leq 10^5$ cm^{-3}) GaN semiconductor layers grown on sapphire substrates through the use of a novel transition structure. The structure consists of a GaN seed layer (grown using a low temperature GaN buffer layer) which is etched to expose sidewalls of GaN from which pendeo-epitaxial regrowth occurs. It has been found that dislocation defects do not significantly propagate laterally in GaN single crystalline films. Furthermore, it has been found that GaN will not nucleate on certain amorphous and crystalline materials (namely silicon dioxide and silicon nitride as well as aluminum nitride and silicon carbide under certain conditions) and certain metals (i.e. tungsten).

Employing these two findings, the GaN seed layers are capped with a mask material and in some areas etched back completely to re-expose the sapphire substrate, thereby forming GaN columnar seed structures. These columnar structures contain two key features. Firstly, sidewalls from which the GaN regrowth can occur without lateral propagation of dislocation defects. Secondly, a mask on the top surface of the columnar structure to prevent vertical propagation of defects by preventing nucleation and vertical growth of GaN from the existing GaN seed columnar structure. Since the areas between adjacent seed structures have been etched back to, and ideally into, the sapphire substrate, nucleation between the GaN seed columnar structures is prevented by choosing appropriate growth system parameters (e.g. temperature, precursor flux, etc.)

This results in the very advantageous ability to grow low-defect density GaN layers over the entire thin film surface in one growth step. Growth can only start by homogeneous nucleation of GaN on the GaN seed columnar structure sidewalls, and growth continues laterally without propagation of the defects present in the GaN seed layer. As the GaN grows laterally, vertical growth on this advancing lateral growth begins, and as soon as the vertical growth exceeds the height of the seed structure mask, lateral epitaxial overgrowth occurs in the opposite direction to the advancing pendeo-epitaxial growth. This results in the practical elimination of all dislocation defects stemming from the heteroepitaxial growth of GaN on sapphire. Once the advancing pendeo-epitaxial GaN growth coalesces with adjacent growth, a complete single-crystal low-defect density GaN semiconductor layer is formed.

9. If not indicated previously, what are possible uses for the Invention? In addition to immediate applications are there other uses that might be realized in the future?

This invention allows for the fabrication of microelectronic and optoelectronic devices requiring a single crystalline low-defect GaN layer. These device layers consist of other III-V nitride components and alloys including AlN, InN, AlGaN, and InGaN. Device structures include, but are not limited to, high electron mobility transistors, field effect transistors, light-emitting diodes and diode lasers, and ultraviolet detectors.

10. Does the Invention possess disadvantages or limitations? Can they be overcome? How?

The sapphire substrate is exposed to the gas phase during regrowth of GaN. Because of the presence of ammonia (NH_3) the exposed areas of the surface of the sapphire are converted to AlN, which is an appropriate material on which can occur the subsequent nucleation of GaN.
This problem can be overcome using a high growth temperature during the regrowth of GaN to reduce the ability of GaN to nucleate on the exposed areas of the sapphire surface.
Another solution to overcome the nucleation problem of GaN on the nitrided sapphire surface is the application of a 'line of sight'-deposition technique such as thermal evaporation or electron-beam evaporation of a masking material, e.g., silicon oxide or a metal, e.g., tungsten. If the mask material is deposited after the etching step, it covers only the top surfaces of the posts and the bottom surfaces of the trenches. The GaN does not usually nucleate on the masks; thus, it is being forced to grow only off the sidewalls of the posts.
A third solution is to etch the trenches sufficiently deep into the sapphire substrate. The lateral growth rate of GaN can be, under certain growth conditions, faster than the vertical growth rate. Under these conditions and with sufficiently deep trenches, the sidewall growth from the posts can coalesce over the trenches before the vertical GaN growth in the trenches resulting out of the nucleation of GaN on the sapphire substrate can interfere with the lateral growth.

11. Enclose sketches, drawings, photographs and other materials that help illustrate the description. (Rough artwork, flow sheets, Polaroid photographs and penciled graphs are satisfactory as long as they tell a clear and understandable story.)

OTHER PERTINENT DATA

1. Is a publication or oral disclosure descriptive of the Invention planned within the next six months? Please give the date (estimate, if known) and attach copies of any existing manuscripts, preprints, abstracts or poster material.

   No publication or oral disclosure planned at this point.

2. Is there an urgency in making a patent application? If so, please give the reason such as, past or future public use of the technology or the likelihood that similar technology may be developed elsewhere.

3. Have any graduate students and/or other technicians been involved in this research? If so, should the individual(s) be included as a co-inventor?

[redacted]

4. Has the Invention been tested experimentally? Are experimental data or prototypes available?

[redacted]

5. Are there any prior applications for patent by the inventor on this subject? If so, give the serial number(s) and the filing date(s).

[redacted]

6. Are there known inventions by other researchers that are related to this one? Please describe, including information on relevant patents and publications, if available.

None Known

7. Was the work that led to the Invention sponsored by industry or funded by State or Federal appropriations? If so, attach a copy of the contract or agreement, if possible, and fill in the appropriate blanks below. One of the below MUST be completed. (This includes the source(s) of funds for the salary of each inventor.)

a. Complete name of government agency:

[redacted]

b. Name of industrial company:
N/A

c. Name of private sponsor:
N/A

d. State or Federal appropriation:
N/A

NCSU 99-79

8. Has the Invention been disclosed to industry representatives? Has any commercial interest been shown in it? Please name companies; listing specific individuals and their titles if you know them.

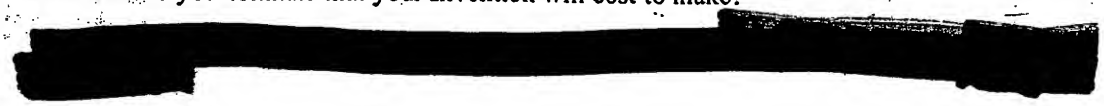
No. Disclosure has not been made to any industry representatives.

a. Do you know of other companies that might be particularly interested in the Invention?

b. List any manufacturers making comparable equipment or products.

None Known

c. How much do you estimate that your Invention will cost to make?



NCSU 99-79

Signature of Inventor(s)

[redacted] (Date)

[redacted] (Date)

[redacted] (Date) [redacted]

Disclosed and Understood by me:

Amy Michel (Witness) [redacted] (Date)

[redacted] (Witness) [redacted] (Date)

[redacted] (Witness) [redacted] (Date)

Department Authorization:

J. M. Risch (Witness) [redacted] (Date)
Department Head/Center Director

[redacted] (Witness) [redacted] (Date)
College Dean/Associate Dean for Research

NC 50 99-79

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Technology Transfer and
Industry Research
Campus Box 7003
1 Holladay Hall
Raleigh, NC 27695-7003

919.515.7199
919.515.3773 (fax)

[redacted]

CONFIRMATION COPY

Mitchell S. Bigel
Myers Bigel Sibley & Sajovec, P.A.,
P.O. Box 37428
Raleigh, NC 27627

Re: "*Process for Growing Low Defect Gallium Nitride Semiconductor Layers on Sapphire Wafers Using Lateral Growth From Sidewalls, and Gallium Nitride Semiconductor Structures Fabricated Thereby*", an invention disclosure by Gehrke, Linthicum and Davis, NCSU file 99-79

Dear Mitch:

This letter serves to authorize you to proceed with preparation and filing of a U.S. patent application for the above-referenced case. [redacted]

Thanks for your help in this matter. Please contact me if you need additional information.

Best regards,

David Winwood, Ph.D.,
Associate Director

Enclosure

cc: Dr. M. Johnson

MYERS BIGEL SIBLEY & SAJOVEC, P.A.

PATENT LAWYERS

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Charles R. Lewis
Karen A. Magri
Robert M. Meeks
D. Scott Moore*
James D. Myers

Timothy J. O'Sullivan
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J. Michael Strickland
John R. Witcher, III

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www.carolinapatents.com

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Licensed in Illinois and Georgia

[redacted]

Dr. Kevin J. Linthicum
North Carolina State University
Materials Science and Engineering Dept.
Box 7919
Raleigh, North Carolina 27695-7919

Re: Patent Application entitled *Pendeoepitaxial Method of Fabricating Gallium Nitride Semiconductor Layers on Sapphire Substrate, and Gallium Nitride Semiconductor Structures Fabricated Thereby*
NCSU File 99-79; Our File 5051-482

Dear Kevin:

Enclosed is an initial draft of the patent application directed to the above invention.

It is essential that the patent application, as filed, be technically accurate and complete, and that it set forth the best mode of carrying out your invention, since new matter may not be added to the descriptive portion after filing. We therefore ask that you and your coinventors carefully review the draft for technical accuracy and completeness and advise us of any suggested changes or corrections. Your changes and suggestions will be carefully considered in the preparation of a final draft, which will be presented to you for your review and execution prior to filing.

We would also like to point out that an inventor is required to make a Declaration when his application is filed in the U.S. Patent and Trademark Office, acknowledging a duty to disclose information of which he is aware and which may be considered to be material to the examination of the application. "Material" in this respect is defined as information that a reasonable examiner would likely consider important in deciding whether to issue a patent.

"Material" information as defined above may possibly include devices, products, publications, etc. which are similar to your invention and which were publicly known before your invention, and it may also include any public disclosure, commercial use, or offer of sale of your invention more than one year prior to the filing date of your application.

If you are aware of any information which you believe might be considered "material," it is vitally important that it be brought to our attention. We can then make a determination as to whether the information should be brought to the attention of the Patent and Trademark Office under the applicable rules.

You should also be aware that certain activities either in the United States or foreign countries prior to filing of the application in the United States may have a bearing on your ability to file corresponding applications in foreign countries under the applicable international treaty. These activities could include public disclosure of your invention in either written or oral form, such as published articles, theses, patents, product announcements and proposals, as well as through commercial exploitation of your invention, including public demonstrations, offers to sell, and sale of products incorporating your invention. If you would like to preserve your right to file corresponding foreign applications on this invention, we recommend that all such activities should be avoided until the U.S. application is on file.

If you should have any questions regarding the matters outlined above, please feel free to discuss them with us.

Best regards.

Very truly yours,

Mitchell S. Bigel

MSB:sef
Enclosure
cc: David W. Winwood, Ph.D. (w/enc.)
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